PHC CWS Analytical Methods Workshop 2001: Participants' Report

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Introduction

The Canadian Ministers of the Environment endorsed the Canada-Wide Standard for Petroleum Hydrocarbons in Soil on May 1, 2001, allowing jurisdictions to begin their implementation activities. Alberta Environment has moved on several fronts including assessment of issues and directions for implementation of the PHC CWS analytical method. This workshop was held in order to review the scientific basis for the PHC CWS and to review the prescriptive and performance based aspects of the analytical method. The workshop also reviewed the role of CAEAL in accreditation and allowed Alberta laboratories and consultants an opportunity to comment on early applications of the method.

The thirty-two people attending the workshop (See Appendix A) on May 31, 2001 in Red Deer, Alberta raised a number of issues. Twelve recommendations were made for the resolution of these issues. This report is intended to capture the will of the participants and provide input for Alberta Environment's consideration when making implementation decisions related to the method.

Workshop Recommendations

Recommendation 1:
No performance-based modification of the CWS method will be accepted until a validation process is in place.

Recommendation 2:
Alberta Environment should lead development of a validation protocol as soon as possible.

Recommendation 3:
Validation criteria for performance-based methods should address soil texture, moisture content and organic matter content.

Recommendation 4:
CCME should contact CAEAL to request proficiency testing samples for the method.

Recommendation 5:
The first CAEAL interlab study should provide an assessment of the method rather than a proficiency test. CAEAL should accredit the method as it is documented in the CWS.

Recommendation 6:
Develop a method for high temperature GC that includes an upper limit for F4.

Recommendation 7:
Procedures should be available for fine and coarse textured soils

Recommendation 8:
Use the Canadian Society of Soil Science method for determination of percent sand.

Recommendation 9:
In order to offer the method to clients now, labs must apply for CAEAL accreditation and must participate in round robin testing when available.

Recommendation 10:
Future drafts of the method should be made available to the public via Alberta Environment and CCME.

Recommendation 11:
The unmodified CCME method should be used for compliance samples while the validation protocols are being worked out.

Recommendation 12:
Analytical decisions should be clearly communicated to clients.

Record of Discussions

Presentation 1: Overview of PHC CWS, Ted Nason, Alberta Environment

Presentation 2: PHC CWS Analytical Method, Richard Turle, Environment Canada

Question:
Is the CCME looking at alternatives to Soxhlet extraction?

Answer:
Performance-based alternatives can be considered if validated. Soxhlet was chosen as the benchmark because it is widely used and proven to be effective at recovering organic contaminants from soil.

Question:
No process is in place for obtaining approval for performance-based methods. Who will review and approve their validation?

Answer:
Labs must validate for their clients and CAEAL. Validation can be carried out in-house.

Statement:
Labs would prefer external review and approval.

General Discussion:
A question was raised on the effect of soil texture on method performance. The broader issue of validation protocols was discussed in more detail later in the program. A question was also raised concerning the use of the CWS method in choosing appropriate site management and remediation alternatives. Regulators will require the method to be used for initial characterization, site closure, and any intervening compliance points. Other methods may be acceptable for tracking remediation process.

Presentation 3: Accreditation Considerations, Arthur Bollo-Kamara, Alberta Environment

Question:
How is uncertainty structured with respect to the PHC method? (i.e., how much uncertainty is acceptable?)

Answer:
No formal protocol developed yet. Several methods can be used and labs should be able to demonstrate that they have run the methods and can produce the results.

Question:
How is ISO defining uncertainty and is it related to accuracy?

Answer:
Uncertainty is defined as dispersion resulting from the method (i.e. precision). It is not directly related to accuracy.

Comment:
Uncertainty is a useful parameter only if accuracy targets are being met.

Response:
CAEAL would like to initiate an on-going round robin for the CWS method. Because of the expense of such a program, at least 20 labs are needed. CAEAL will assess labs against Standard Operating Procedures, performance samples, and a site visit.

Comment:
A concern was raised about the use of spiked samples. The speaker felt that real samples drawn from contaminated sites were more difficult to work with and would be a better test of method and laboratory performance.

Response:
The advantages of real-world samples was acknowledged but gathering the samples and characterizing them adds significant expense.

Comment:
A concern was raised that the detection limit for F1 was too close to the most conservative guideline value.

Response:
This will be easier to respond to as we gain experience with the method. We will have to see how the method works in actual practice. Small details in the method are very important. Each lab should establish its own uncertainty.

Question:
Is sample heterogeneity accounted for in the uncertainty estimates?

Answer:
It can be, depending on the method used.

Presentation 4: Chris Swyngedouw, Norwest Labs

Comment:
Approvals and other regulatory documents specify analytical methods. The approval holder must ask the Director for permission to use alternatives.

Question:
Who validates modifications - CAEAL or Alberta Environment?

Answer:
CAEAL does not validate methods, only confirms scientific validity. Regulators must accept the modification where approvals or other regulatory documents specify a method. It is the client (approval holder) who must obtain Alberta Environment's acceptance.

General Discussion:
A concern was raised that there is no process in place to approve modifications, except for the laboratory's internal process, which will be audited by CAEAL eventually. Would like to see a process for full verification. Proposed limiting CWS method to prescriptive elements only until a process for regulatory validation is in place.

Presentation 5: Wayne Rae, Maxxam Analytics

Environment Canada Comments:
Some aspects of the method, such as holding times, are the result of practical compromises. If the method is very prescriptive it may become uneconomic. Performance based methods have worked well for other substances, for example dioxins. The Analytical Methods Technical Advisory Group targeted calibration and columns because these were responsible for the most variation.

With respect to high temperature GC, the gravimetric heavy hydrocarbon method was a compromise because many labs do not have the capability to reach C50. Did not include a method for high temperature GC because they could not find any published methods to evaluate. This led to an ongoing discussion of other issues such as laboratory uncertainty versus other uncertainties (e.g., soil heterogeneity).

Development of Recommendations

The following discussion focussed on developing a list of recommendations.

Policy for Implementation of Performance-Based Alternatives

Recommendation
No modification of the CWS method should be accepted until a validation process is in place.

Validation Process for Performance Based Methods

Question:
What is the validation process if a new method is developed?

Answer:
CAEAL accreditation is essential but additional work is necessary because of method complexity. Alberta Environment should develop a validation protocol; laboratory consensus will be needed.

Question:
Why not allow validation of a totally different method (i.e., including changes to prescriptive elements)?

Answer:
This issue is better served by CCME rather than by individual jurisdictions. CCME has a review process every 5 years to make revisions.

Comment:
In the PHC Standards, hexane and acetone solvent is a benchmark, but we are getting good results with the shake and dichloromethane (DCM) method.

Response:
We have to avoid DCM due to health risks to lab staff. As hexane and acetone are used, we will get more information. Have to stick with hexane and acetone now. We have to find a way around using silica gel too. If there is something better to use bring it forward.

Recommendation
Develop a validation protocol as soon as possible. Consensus will be necessary.

Performance Testing

General Discussion:
A round-robin should be organized by CAEAL as soon as possible. CCME should contact CAEAL to coordinate this. CAEAL needs at least 20 labs nationwide to cover the costs of the program. First round will be a learning exercise. We want to have enough samples to use prescriptive methods and the lab's own performance based alternatives to see how closely they align. When we contact CAEAL we will request the number of samples to prepare.

Recommendation
CCME should contact CAEAL to request performance testing samples for the method.

Recommendation
Next CAEAL interlab study should provide sufficient sample material to allow performance-based calibration.

CWS Method Improvements

Recommendation
Develop a method for high temperature GC.

Recommendation
Procedures should be available for fine and coarse textured soils

Recommendation
Use the CSSS method for determination of percent sand after extracting hydrocarbon.

CAEAL Accreditation Process

General Discussion:
What is Alberta's standpoint on accreditation? Can we start using CCME PHC methods even if we are not accredited to use them yet?

One lab received a letter from CAEAL indicating there is a 24 month rollout process for the CCME PHC Standards.

Labs have to be accredited now and then within 2 years get accreditation for PHC.

CAEAL accredits for processes. Their scheduling is tight: you can't get accreditation in 2001 if you apply now, have to wait.

Labs need 24 months for review and accreditation - 2 year cycle.

Lab must be "in the stream" for CAEAL accreditation.

If process takes 2 years then no labs can process soil samples for PHC.

Only 1 lab has received accreditation to do PHC - Envirotest Labs.

Normally a lab would be visited every 24 months and audited and participate in PT when it is available. Lab must be "in the stream" for CAEAL accreditation and must take part in PT when implemented.

Recommendation
In order to offer the method to clients now, labs must have applied for CAEAL accreditation and must participate in round robin testing.

Communications

General Discussion:
Communicating new CCME developments is being done poorly. Labs are getting information through clients.

Each member of the technical advisory group sends out information, so it isn't widely available. In the future, this information may be available on the website.

Translating the information to French was too expensive, so information has not been disseminated properly.

Information can be added to provincial website to get around the language issue.

Recommendation
Future drafts of the method should be made available to the public via Alberta Environment and CCME.

Reporting

General Discussion:
How to report C34-C50? As C50+? C50? C34+?

Need a definite upper carbon value. Determining its value should be part of method development.

There is no intent to capture hydrocarbons in asphalt. A review of hydrocarbon properties in crude oils and flare pits should be conducted.

The type of hydrocarbon ties in with the aging of hydrocarbons.

The decision to use C50 came from a general consensus that this was the upper limit of quantitation for conventional chromatography.

Most data doesn't include values greater than C50.

Here in Alberta there are C60 and C70 hydrocarbons.

Question:
Federated Crude was used in the CCME study. How high did the hydrocarbons go?

Answer:
Not sure, but about 24% by mass of the crude oil was in F4.

Recommendation
Develop an upper limit for F4 in the high temperature GC method.

Using Other Methods to Analyze Hydrocarbons

General Discussion:
Other methods have validity for use; use PHC method only for site closure.

Use another method to find out what is there. Other points in time in other situations may need PHC data before the time of closure, e.g. for sites under risk management.

Initial characterization could be done using other methods.

Soxhlet extraction, silica gel and GHH should be used for compliance samples until performance-based alternatives are validated.

Recommendation
The unmodified CCME method should be used for compliance samples while the validation protocols are being worked out.

Validating Performance-Based Aspects of CWS Method

General Discussion:
Validation process - shake with DCM is 100% recovery and shake with hexane and acetone in *clay* soils is only 50% recovery, so there is a huge difference.

This is something to discuss in the lab.

DCM extracts polar organics out of the soils as well.

Hexane and acetone take out polar organics too.

When validating the extraction method, it was found to work well with most soil textures, moisture contents and organic contents.

Recommendation
Validation criteria for performance-based methods should address soil texture, moisture content and organic matter content.

Service to Clients

Question:
What about educating clients?

Answer:
All labs and AENV have to educate the public. Clients holding operating approvals from AENV should be aware of the methods specified in the approval.

Comment:
Turn around time could be a problem. Another faster extraction method would be better. Results were obtained 5 times faster before.

Response:
No, actually the results were obtained about 2 times faster before.

Comment:
We need a faster method when we have equipment in the field. It's very expensive to wait for the results.

Response:
Do duplicate analyses. We can't have instantaneous results, have to compromise.

Question:
How do we know how many samples to take, where to sample? Is there a standard number of samples per cubic meter?

Answer:
With more expensive testing, you increase your costs, decrease the number of samples, and therefore, decrease the amount of statistical data you have. With a fixed budget you will have to reduce the number of samples you submit for analysis. Best to communicate with the lab so that you can provide good recommendations to clients.

Question:
How many samples are enough?

Answer:
AENV does not have anything prescriptive on this and is not developing anything for Phase 2 site assessments. Contaminant release scenarios are so diverse that any prescriptive guidance is bound to fall short in certain situations.

Question:
Clients of labs ask for advice from lab personnel. Should only a professional chemist or engineer sign the report?

Answer:
Develop a dialog with the analyst. This can assist the consultant in making a balanced recommendation on numbers and locations of samples.

Question:
Would providing background information on a site on the chain of custody form be helpful for the labs?

Answer:
That information would be helpful. It is desirable to put background information on the chain of custody form, but it is up to labs to decide if they want the information.

Recommendation
Analytical decisions should be clearly communicated to clients.

Appendix A - Meeting Attendees

Consultants	
Kathryn Bessie	EBA Engineering Consultants
Geraint Edmunds	Envirisk
Francisco Fernandez	UNOTEC
Kelly Gough	Keystone Environmental
Ari Laurell	UNOTEC
Ron Lincz	Newpark Environmental Services
Chris Powell	Komex International
Trish Snethun	ARC Inc.
Miles Tindall	Komex International
Laboratories	
Brenda Chomin	AMEC
Bob Corbet	Access Labs
James LeBlanc	AMEC
Koshy Malayil	Kaizen
Andrew Masters	Maxxam labs
Ron Minks	Envirotest
Wayne Rae	Maxxam labs
Elke Romahn	Agat Labs
Chris Swyngedouw	Norwest labs
Regulators	
Karen Blank	National Energy Board
Arthur Bollo-Kamara	Alberta Environment
Gordon Dinwoodie	Alberta Environment
Jock Forster	Alberta Environment
Jim Fujikawa	Energy and Utilities Board
Sue Halla	Energy and Utilities Board
Darlene Howat	Alberta Environment
Jamie Kereliuk	Alberta Environment
Ted Nason	Alberta Environment
Bill Pelech	Alberta Environment
Richard Turle	Environment Canada
Femke Want	Alberta Environment
Kelly Young	Alberta Environment
Mike Zemanek	Alberta Environment